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FOURTH INTERIM REPORT

R & D project No. 5647-MS-01
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Title: Martensitic Phase Transitions in Nanocrystalline Materials

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As indicated in our third interim report, we have carried out Mössbauer spectroscopy on isolated nanometer-sized crystals cooled to 4.2 K under ultra-high vacuum conditions in order to decide whether or not nucleation of martensite is possible in nanometer-sized, unconstrained crystals.

The spectrometer necessary to carry out those experiments was developed in January 1988. It consists of a quartz tube which has locally a section of two thin walls which are transparent to the Mössbauer (gamma)-rays. The loose powder of isolated small crystals is filled under UHV conditions into the tube and is subsequently cooled to 4.2 K. However, when we recorded the Mössbauer spectra, they turned out not to be conclusive for two reasons.

(1) The isolated 5 nm Fe-Ni crystals are superparamagnetic at 4.2 K. (2) The resolution of the spectra is not as high as required to distinguish between bcc and fcc FeNi due to the small amount of material in the tube. Iron, Nickel (alloys)

In order to keep the crystals isolated,the density of the powder in the quartz tube had to be less than 1 % of the bulk density of Fe-Ni. This low density results in a weak (Mössbauer) spectrum with poor resolution.

Since January, several ideas have been tried to overcome these limitations. However, we had little success. Our last attempt involved a redesigned tube. Nevertheless, the result was still not satisfactory.

We have now decided to give up on Mössbauer spectroscopy and apply low temperature X-ray studies of isolated small Fe-Ni crystals. The small crystals are embedded in a protective layer of paraffin. This layer prevents oxidation. If martensite would form in the small crystals upon cooling, it can be detected due to its tetragonal lattice structure.

The embedding procedure has been tested successfully a few days ago. Therefore, it seems likely that we can carry out the low temperature X-ray measurements (at 4.2 K) in the near future. The KFA Jülich has a low temperature X-ray stage which will be made available to us for these measurements.

Note added in November 1988

The planned X-ray experiments mentioned in the third section of this page have been partially carried out. Isolated small crystals (5 nm diameter) of an Fe-Ni-alloy coated with a protective layer of paraffin have been prepared. About 70 % of the crystals had γ-Fe structure; 30 % were transformed martensitically (α-Fe). Both phases showed up in electron microscopy and in the X-ray diffraction diagrams recorded. When the specimens were cooled from 293 K to 4.2 K followed by a subsequent reheating to ambient temperature, no variation of the volume fraction of martensite was noticed.

This result may be interpreted in the following two ways. (i) The volume fraction of martensite remained constant at all temperatures. (ii) The volume fraction changed upon cooling. However, during the subsequent heating, the martensite formed transforms back into γ-Fe.

It is planned to distinguish between both interpretations by recording diffraction diagrams at 293 K, 4.2 K and again 293 K. When this data is available, it should be possible to answer the question how martensitic phase transitions occur in nanocrystalline materials. This was the goal of the proposal. The study is expected to be finished by December 1988 / January 1989.